Role of USAXS in Advanced Energy Materials Characterization – Past, Present, Future

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Ultrasmall-angle x-ray scattering (USAXS) has formed an integral part of the SAS capabilities at APS since 1998. Absolute intensity-calibrated USAXS measurements enable microstructural features to be characterized and quantified over a contiguous length scale range from nanometers to micrometers [1]. This makes the technique particularly suited to the study of materials containing hierarchical void structures over many length scales such as occur in complex energy materials. When combined with the x-ray brilliance of a third-generation synchrotron undulator, the USAXS technique can cover much of this scale range within a single measurement using a beam size (hence spatial resolution) down to a few micrometers in one dimension, making USAXS suitable for interrogating gradient microstructures such as occur in gas separation membranes or solid oxide fuel cell (SOFC) systems. Furthermore, USAXS can be used in a 2D-collimated configuration with arbitrarily high azimuthal angular resolution for characterizing materials containing extremely anisotropic microstructures such as thermal barrier coatings. Meanwhile, the tunability of the undulator provides great flexibility in the choice of x-ray energy, not only enabling the study of thicker, more representative samples, but also enabling anomalous USAXS studies to be made of the electrochemically-active interfaces found in fuel cell systems.

While USAXS studies contribute to materials research across all materials classes, the role of USAXS measurements in advanced energy materials research has been, and remains, particularly prominent. This point will be demonstrated by a brief review of our extensive USAXS characterization of advanced thermal barrier coatings used in gas turbines, and by presenting recent anomalous USAXS measurements of the electrochemically active interfaces in SOFC systems.

Currently, a 2D pinhole small-angle x-ray scattering (SAXS) measurement capability is being incorporated into the USAXS instrument; this will enhance the sensitivity to nanoscale features, and will enable small-angle diffraction measurements to be combined with calibrated USAXS/SAXS materials microstructure characterization. Plans to upgrade the USAXS beam optics to extend the x-ray energy range to ≈ 30 keV will both enhance the capability to combine USAXS, SAXS, and diffraction, and will allow the use of thicker window materials for *in situ* characterization chambers. The potential of these developments for future *in situ* studies of gas separation membranes and carbon capture sorbent materials will be outlined.

[1] J. Ilavsky, P.R. Jemian, A.J. Allen, F. Zhang, L.E. Levine and G.G. Long; "Ultrasmall-angle X-ray scattering at the Advanced Photon Source," with supplementary material, *J. Appl. Cryst*., 42, 469-479 (2009).